10.0 MAINTENANCE

10.1 Obtaining Service:

All servicing should be performed by a Perkin-Elmer service representative. During down time the instrument will be tagged "out of service" and lock out / tagout procedures will be followed (See Policy and Procedure manual for tagout procedures).

10.2 Daily Care:

10..2.1 Cu ABS

The instrument will be adjusted prior to analysis with the Cu lamp. The final Cu ABS will be recorded in the Flame Maintenances and Repair Log.

10.2.2 Cleaning the Burner Head

The burner should provide an even flame over the length of the burner head slot. An uneven flame may indicate the slot needs cleaning. Proceed as follows:

- Extinguish the flame, allow the burner head to cool, shut down the gases at source, and bleed the gas supply lines
- Remove the atomizer compartment door
- Remove the burner head
- Rinse the burner head well with deionized water and make sure that all scrapings are removed from the inside and outside the burner head
- Blow the burner head dry with clean compressed air
- Reinstall the burner head

10.2.3 Cleaning the spray chamber

- Extinguish the flame, allow the burner head to cool, shut down the gases at source, and bleed the gas supply lines
- Remove the burner head
- Pour a large quantity of deionized water slowly through the neck of the spray chamber to thoroughly flush the inside and the drain system
- Remove the nebulizer
- Disassemble the spray chamber
- Clean the spray chamber and flow spoiler with a soft brush and a mild laboratory detergent solution
- After cleaning rinse thoroughly with deionized water
- Reassemble the burner system

10.2.4 Reassembling the burner system

See Figure 10.2

- Place the spray chamber back onto the burner carriage
- Locate the two milled posts into the threaded holes in the carriage and then tighten the two milled posts firmly to secure the spray chamber
- Slide the cleaned flow spoiler fully back into the spray chamber such that one of the blades is in the "six o'clock" position
- Check that the burner end cap O-ring (or gasket) is properly seated in the groove inside the end cap and is in good condition. Replace if necessary
- Place the end cap against the spray chamber and tighten the four thumbscrews. Thighten them in an alternate pattern until they are finger-tight
- If you removed the auxiliary oxidant and fuel hose, reconnect them: Tighten the compression fittings hand-tight plus a 1/4 turn using the double-wrench technique (hold the end cap fitting with one wrench and tighten the compression fitting with the other wrench)
- If you removed it, carefully push the drain tube onto the drain fitting and tighten the clamp
- Plug in the drain interlock connector and secure it with the securing ring
- Check that the burner head O-ring is properly seated in the neck of the spray chamberand is in good condition. If it is not, replace.

  Note: Apply a very small amount of Apeizon grease to the inner surface of the O-ring in order to form a good seal with the burner head. Do not let any of the grease get inside the chamber. If you do, wipe the chamber surfaces clean with a dry cloth.

- Place the retaining ring back on the spray chamber and hand tighten
- Reinstall the burner head

10.2.5 Maintaining the drain system

Always place the drain vessel in a well ventilated place underneath the spectrometer, in full view while you are working with the spectrometer. This prevents the build-up of potentially hazardous gases, and allows you to see the liquid level.

You must check the drain tube and vessel periodically for wear and/or chemical atttack. The drain tube should be clear with as few bends as possible. You should replace the drain tube if it has deteriorated.

10.2.5.1 Flushing the drain system

- Extinguish the flame, allow the burner head to cool, shut down the gases
- Remove the atomizer compartment door
- Remove the burner head
- Pour a large quantity of deionized water slowly through the neck of the spray chamber to thoroughly flush the inside and the drain system
- Empty the drain vessel

10.2.5.2 Emptying the drain vessel

- Extinguish the flame, allow the burner head to cool, shut down the gases
- Switch off the spectrometer
- Flush the drain system
- Unscrew the retainer cap from the drain vessel
- Carefully lift out the drain trap assembly. Do not disconnect the electrical lead
- Place the drain trap assembly into a suitably large bucket or container so that it is vertical. If the drain tap assembly is laid on its side, liquid can run out onto the floor
- Rinse the outside of the assembly well with water
- Empty the drain vessel
- Place the drain trap assembly back into the drain vessel and screw on the retainer cap

10.2.5.3 Adding water to the drain trap

Enough water must be present in the drain system to fill the drain loop and to activate the drain float. If not, carry out the following procedure:

- Extinguish the flame, allow the burner head to cool, shut down the gases at source
- Remove the atomizer compartment door
- Remove the burner head
- Pour about 250 mL deionized water slowly through the neck of the spray chamber to fill the drain trap and activate the drain float
- Reinstall the burner head
- Refit the atomizer compartment door

10.2.5.4 Cleaning the drain trap assembly

The drain trap assembly is located inside the drain vessel. Periodically you should remove the assembly and clean it.

- Extinguish the flame, allow the burner head to cool, shut down the gases at source
- Switch off the spectrometer

- Flush the drain system
- Unscrew the retainer cap from the drain vessel
- Carefully lift out the drain trap assembly. Do not disconnect the electrical lead
- Place the drain trap assembly carefully with mild laboratory detergent solution using a test tube brush. Make sure that the float moves freely
- Rinse the assembly well with water
- Empty the drain vessel
- Place the drain trap assembly back into the drain vessel and screw on the retainer cap

10.2.6 Maintaining the acetylene supply

- Acetylene is supplied dissoved in a solvent such as acetone, and a small amount of solvent carryover with the acetylene is normal. However, as tank pressure falls, the relative amount of solvent entering the gas stream increases and can give erratic results. For this reason, replace acetylene cylinders when the cylinder pressure drops to about 100 psi.

10.3 Monthly

10.3.1 Maintaining the nebulizer

To maintain optimum nebulizer performance you must periodically clean the capillary assembly, and occasionally replace the sample tube and the PTFE tube in the capillary asembly gland nut.

When you aspirate concentrated samples with complex matrices, it is especially important to keep the spray chamber and nebulizer free of deposits. It is beneficial to periodically aspirate a dilute surfactant solution, such as 0.1% Triton X100, for several minutes to keep the system clean. The surfactant will encourage constant drainage, which is essential for optimum burner system performance.

Before you clean the nebulizer, because you suspect that the nebulizer is causing low absorbance readings or poor sensitivity check that:

- The sample tube is in good condition (i.e. no kinks), is not too long, and is clean. With a longer sample tube, the rate of sample uptake, and thus the sensitivity, will decrease. Recommended length: 20 cm
- The burner head is clean
- The burner is correctly optimized

10.3.2 Cleaning an obstruction in the nebulizer

If you suspect that there is an obstruction in the nebulizer, try to clear it as follows:

- Remove the nebulizer from the burner end cap (See Figure 10.2)
- Remove the sample tube from the PTFE tube protruding from the end of the nebulizer
- Take one of the copper cleaning wires, provided with the nebulizer, and very carefully push it through the PTFE tube and out through the sapphire tip. It may take several attempts to pass the wire through the narrow sapphire tip. Move the wire in and out to dislodge any particles
- Insert the sample tube into the PTFE tube protruding from the end of the nebulizer until it is held firmly, **but is not bent or crimped**
- Reinstall the nebulizer
- Relight the flame and then aspirate pure solvent to rinse the nebulizer

10.3.3 Removing the nebulizer

- Extinguish the flame, allow the burner head to cool, shut down the gases at source, and bleed the gas supply lines
- Remove the atomize compartment door
- Lift up and hold the nebulizer clamp. Very gently rock the nebulizer a few times to make sure that it is not sticking in the end cap
- Carefully turn the nebulizer counterclockwise until the side arm is horizontal in the side arm slot. This releases the bayonet lock

Note: Do not apply force to the side arm since you could otherwise break it off

- Carefully pull the nebulizer out of the end cap
- Unscrew the side arm nut and carefully withdraw the nebulizer oxidant tube from the side arm with a rocking motion

10.3.4 Cleaning the nebulizer

After you have disassembled the nebulizer, clean the components as follows: (See Fig 10.3)

- Gently pull the PTFE tube out of the gland nut, using needle-nose pliers if required
- Clean the capillary assembly, the gland nut, and the PTFE tube using a mild laboratory detergent solution, or a solvent such as isopropanol. If the PTFE tube or the gland nut still appear to be dirty, discard them and replace them with new
- Clean the nebulizer body and any other parts that appear dirty in a mild laboratory detergent solution
- Rinse all the cleaned components thoroughly with deionized water
- Inspect the O-ring and clean them as necessary or replace if necessary

10.3.5 Reassembling the nebulizer

- Insert the PTFE tube into the threaded end of the gland nut until it is just held in the gland nut. If you are replacing the PTFE tube, cut a 23 mm (0.95 in.) length
- Screw the gland nut into the capillary assembly until it becomes hard to turn. This forces the PTFE tube back into the gland nut and ensures that the tube is properly seated inside the capillary assembly
- Replace the sample tube:
  1. Cut a 20 cm length from the 0.6 mm i.d. tube
  Cut the tube at a 45 degree angle so that is can easily be inserted into the nebulizer
  2. Press and rotate a tapered push-pin or similar into the PTFE tube protruding form the rear of the gland nut. This will expand the opening in the PTFE tube and facilitate insertion of the sample tube
  3. Insert one end of the sample tube into the expanded PTFE tube until it is held firmly, but is **not bent or crimped**
- Slide the spring back onto the capillary assembly
- Inspect the tiny O-ring on the capillary assembly
- Slide the assembled capillary assembly into the nebulizer body:
  1. Line up the alignment key with the keyway in the nebulizer body
  2. Push the capillary assembly in until it bottoms out in the nebulizer body
  3. If you inserted the capillary assembly correctly, it should move freely in and out of the nebulizer. If it does not, remove and reinstall, paying particular attention to the alignment key
- Screw the locking ring onto the nebulizer body (if it was removed)
- Screw the regulator onto the nebulizer body

Reassembling the spray chamber

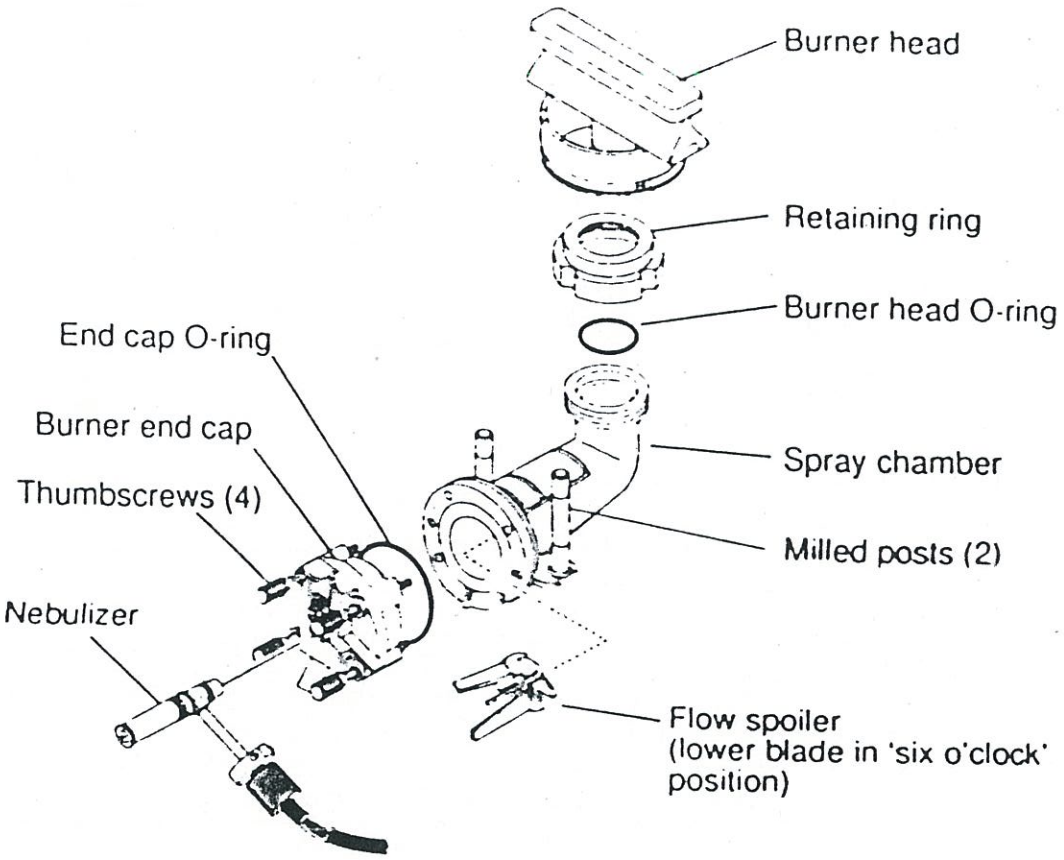


Figure 10.2 Exploded View of the Burner System

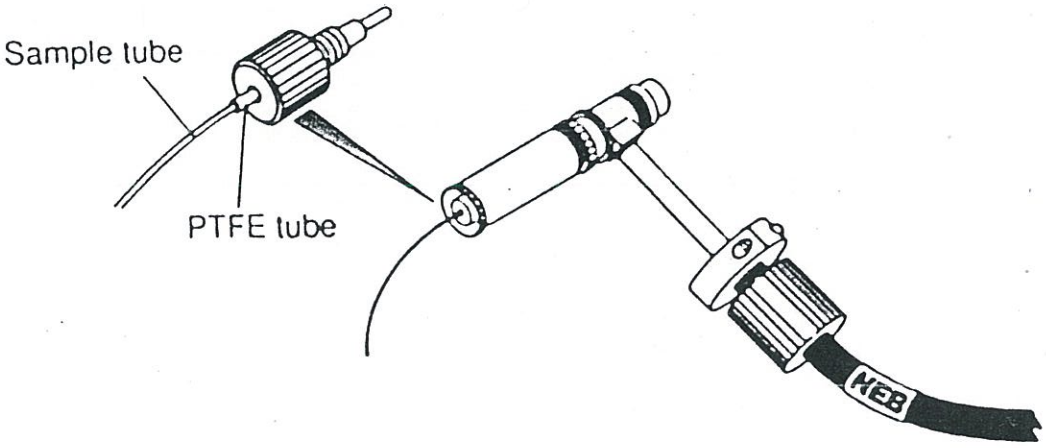


Figure 10.2 Nebulizer Sample Tube and PTFE Tube

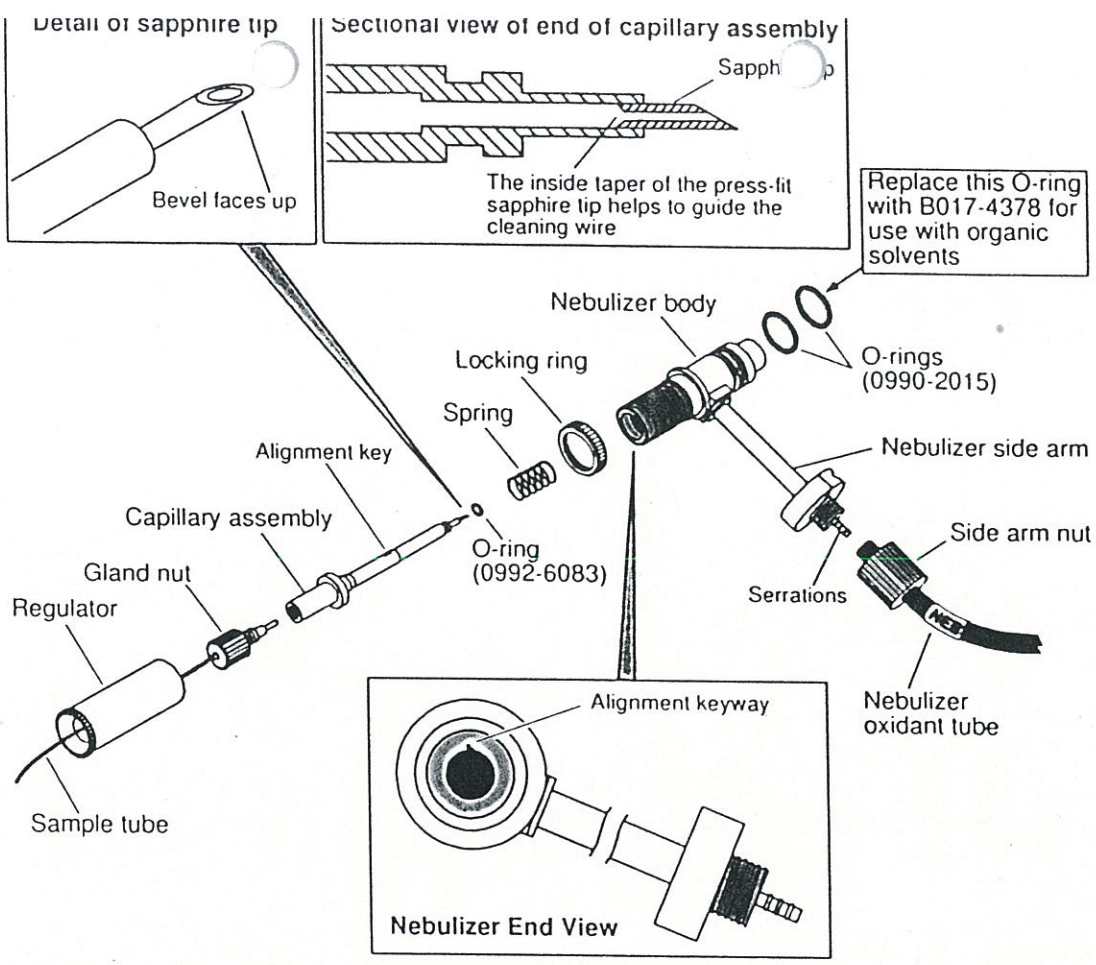
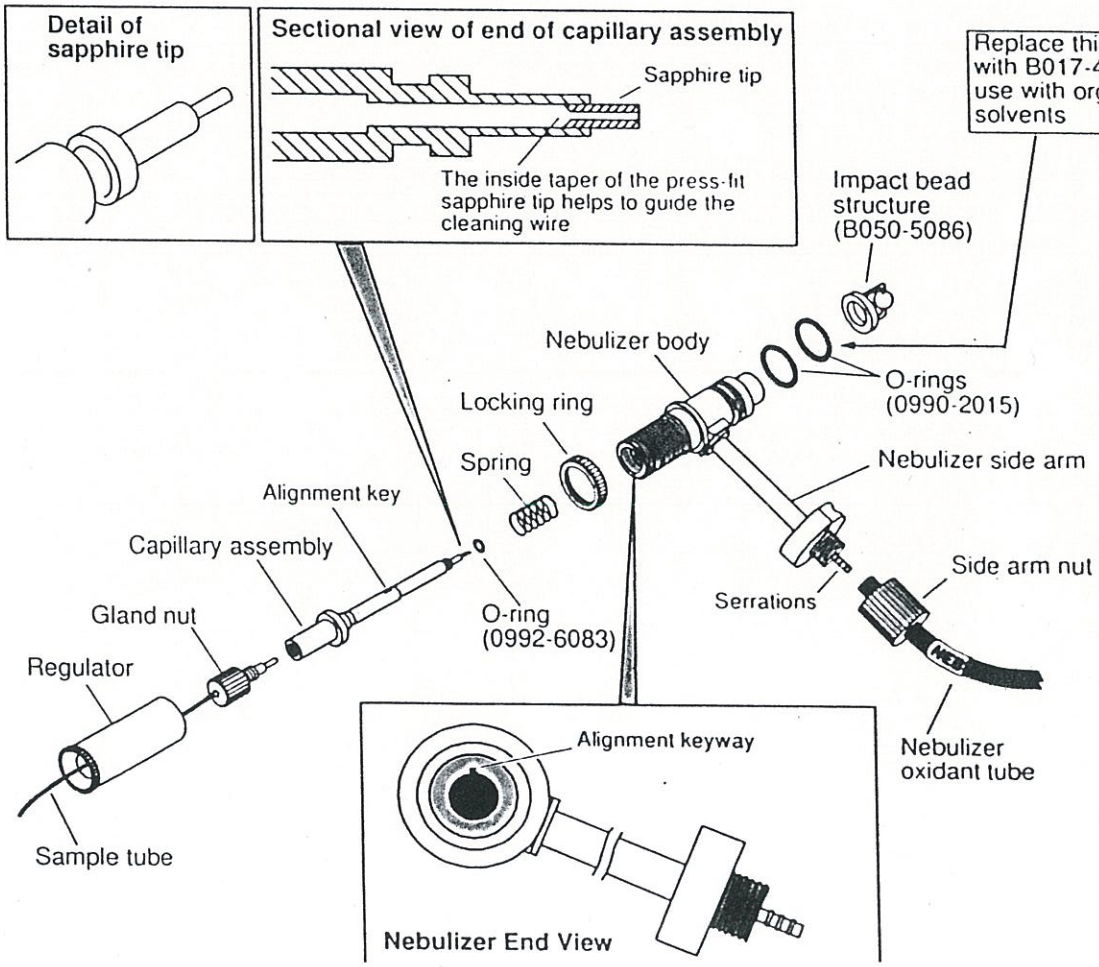


Figure 10.3 Standard GemTip Nebulizer Assembly (N037-0394)



11.0 TROUBLESHOOTING

11.1 Service

The following suggestions are included to help the operator determine systematically whether difficulties experienced with the spectrometer are due to a malfunctioning of the instrument, improper settings of the instrument controls, or improper analytical techniques.

If after checking the instrument against the symptoms described below, the source of the difficulty appears to be due to a malfunctioning of the instrument, it is strongly recommended that the operator of the instrument report to the immediate supervisor. The supervisor will then contact a Perkin Elmer service representative. All lockout/tagout procedures will apply.

11.2 Hollow Cathode Lamp:

11.2.1 Lamp will not light

- Make sure the instrument is plugged in, turned on, and warmed up.
- Check other lamps to determine if the failure to light is characteristic of the instrument or of the individual lamp.

11.2.2 Lamp current cannot be set to the desired operating value.

- If the lamp formerly could be run at the proper current, inability to reach the desired current setting can be an indication that the lamp is starting to deteriorate.

11.2.3 Align Lamps window:

- The Counts values shown in the Align Lamps window are higher than those shown in previous versions of AA WinLab. This is because the value for the sample beam is shown whereas previously the value for the reference beam was shown. The actual sample beam intensity is the same as in previous versions of AA WinLab.

11.2.4 EDL's current settings

- For some EDL's the default current setting that the instruments automatically use is sometimes too high. If the lamps switch off automatically or if the Counts

value -- shown in the Align Lamps window, exceeds 30000 reduce the current (for example in 10% steps) until the Counts value is below this value.

11.3 Change Technique

- If you select a different technique, then cancel the procedure while the system is changing, the system may stop in an undefined status. To make sure that the system is correctly set up, change the technique again to that which you require.

- With an AAnalyst 800, before you change the technique from Furnace to either Flame or Furnace, or vice versa, switch off EDL's. When you have changed the technique, make sure that the lamp current is set correctly in the Align Lamps window.

11.4 Multi-method analyses

- If you have selected more than one method in the Automated Analysis: Setup window, and these methods use different lamps, always select the option to switch off the lamps at the end of the analysis **Off after analysis - Lamp**. The system will switch off the lamp when it has finished using a method, and will switch on the lamp it needs for the next method.

- If you print the autosampler loading list, AA WinLab may hang up.

- Interrupting Multi-method analyses
  If you interrupt a multi-method analysis and then restart it, the dialog "Continuing an Analytical Sequence" does not always appear. Instead, the analysis starts again with the first sample.

11.5 Flame Control Window

- The function key F10 to ignite and extinguish the flame works correctly only if the Flame Control window is open. However, if a sub-window is open in the Method Editor, in the Flame control window, the Flame On/Off switch is disabled, and the control window, the Flame On/Off switch is disabled, and the function key F10: extinguish the flame, does not work. In an emergency, switch off the instrument using the on/off switch. Otherwise, close the sub-window to activate these controls. A sub-window is one that contains OK, Cancel and Help buttons.

11.6 Reformat utility

- Before you start the Reformat utility, close all windows in AA WinLab.

11.7 Software Errors

- A message with a number is displayed. Most problems of this type are simple procedural errors. You may try to access a library in a location where none exists or the system is unable to create a directory for some reason. It should be possible to solve these common problems from the information contained in the message that is displayed. If you need more information for Library Manager messages, see the ***Messages*** topic.

- Two programs try to access a library at the same time. The Library Manager needs exclusive use of libraries to perform certain operations such as packing libraries, checking libraries, or deleting data sets. Sometimes lock files are left behind when a program terminates unexpectedly. Usually the solution to these problems will be evident form the messages displayed. In some cases you may need to use the File Manager to remove files with a .lck extension from the directory containing the library tables. If you are unable to solve the problem easily, try exiting and restarting Windows.

- Operations become very slow. It is possible for data saving and retrieval operations to become slow if the results library becomes excessively large. The solution to this problem is to periodically archive results data sets, delete them from the library, and then pack the library as discussed under the ***Recommended Library Maintenance Procedures*** topic.

- Serious library errors occur. Problems of this type typically mean that one of the library tables has become damaged or is missing for some unknown reason. The AA WinLab software will display a message dialog in which the first line states A serious result library error has occurred (or method library). The first thing to try is to exit the application and restart Windows. If that does not fix the problem try using the check function in the Library Manager to examine the library. If error are found that the check function can not repair, you will have to create a new library or replace the damaged library with a backup copy. The topic on ***Checking A Library*** describes how to use this function.

11.8 Results Questionable:

11.8.1 Results Erroneously High

A) The hollow cathode lamp warm-up time insufficient.
B) Standard solutions improperly made.

11.8.2 Results Erroneously Low

A) Check lamp warm-up time.
B) Chemical or matrix interferences.
C) Standard solutions are improperly made.
D) Blank solution is contaminated.

11.8.3 Cannot Get Suggested Detection Limits or Close to Suggested Sensitivity

A) Insufficient scale expansion and integration times are being used.
B) The sensitivity for analysis is too low.
C) The lamp current is too low for desired stability.
D) The analysis is being performed on the wrong spectral lines.

11.9 Results Noisy:

A) Check lamp warm-up time. Lamp should be warmed for at least 45 min.
B) Check alignment of the lamp.
C) Check wavelength

12.0 Waste Disposal

All waste disposal procedures will follow the Water Quality Laboratory Chemical Hygiene Policy and Procedure Manual (sections 3.11.0 and 1.0.0). The following applies for sample disposal procedures:

A) Discard all remaining analyzed samples in an acid sink.
B) All disposable containers will be rinsed with tap water and disposed of in trash.

13.0 TRAINING

OFFICAL DOCUMENT

13.1 When analysts are first introduced to this procedure, a current copy of the SOP for this instrument will be provided to the analyst. Prior to performing this procedure the analyst will study this SOP in detail to assure complete acquaintance with this procedure and be tested before starting any independent analyses.

14.0 REFERENCE MANUALS

14.1 Perkin Elmer Atomic Spectrometry Aanalyst 800

14.2 Perkin Elmer Atomic Spectrometry THGA Graphite Furnace

14.3 Analytical Method For Atomic Absorption Spectrophotomet

14.4 Perkin Elmer Atomic Spectrometry Burner System - User's Guide

14.5 City of Albuquerque Water Quality Laboratory Policy and Procedure Manual

14.6 City of Albuquerque Water Quality Chemical Hygiene Policy

15.0 ANALYST NOTES

MRI 6-19-02 MODIFIED SECTION 8.1.15 TO REQUIRE +/- 5% FOR ICVS & CCVS

CAN 6-20-02 ±5% for Drinking Waters. Changes in ICP/FURNACE ONLY.

ALL BATCHES ARE TO BE RUN WITH AN INITIAL CCVS m. 12/20/04

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DATE 4/9/09
QA